

# Synthesis and Characterization of Barium Titanate ( $\text{BaTiO}_3$ ) Nanoparticle

M.R.A. Bhuiyan, M.M. Alam, M.A. Momin, M.J. Uddin, M. Islam

Department of Applied Physics, Electronics and Communication Engineering, Islamic University

Kushtia 7003, Bangladesh

Email: [mrab\\_iu@yahoo.com](mailto:mrab_iu@yahoo.com) (M.R.A. Bhuiyan)

**(Abstract)** Barium titanate ( $\text{BaTiO}_3$ ) nanoparticles were synthesized via an electrochemical route from Ti metal plate at room temperature. Structural, compositional and optical properties were characterized by XRD, SEM, EDX, FTIR, UV-Vis and photoluminescence (PL) spectroscopy. X-ray diffraction (XRD) confirmed the preferential growth of  $\text{BaTiO}_3$  nanoparticles that width is  $\sim 15$  nm in the (110) orientation. The SEM image shows the synthesized  $\text{BaTiO}_3$  were nanowires in shape. The EDX measurements confirm that the composition of the samples was Ba, Ti and O elements. UV-Vis Spectroscopy shows the absorption peak at  $\sim 330$  nm. PL measurements reveal an intense and broad band at around the green colour emission region.

**Keywords:** electrochemical,  $\text{BaTiO}_3$ , nanowires, structural and optical properties

## 1. INTRODUCTION

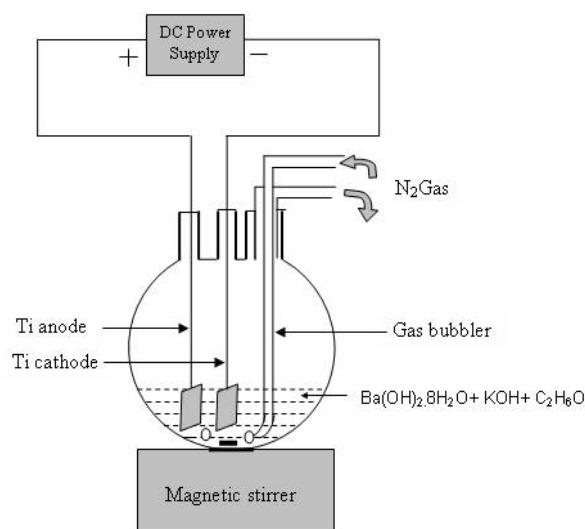
$\text{BaTiO}_3$  have been attracted considerable interest for a wide variety of applications. It has a perovskite structure that possesses a high dielectric constant [1] and widely used in multilayer ceramic capacitors (MLCCs), dynamic random access ferroelectric memories (DRAMs) [2, 3].  $\text{BaTiO}_3$  nanoparticle have been synthesized by using hydrothermal method, sol-gel processing, composite-hydroxide-mediated method, flame assisted spray pyrolysis (FASP), radio-frequency plasma chemical vapor deposition (RF-plasma CVD), one step sol-precipitation route and electrochemical route [4-10]. In hydrothermal process to synthesized  $\text{BaTiO}_3$  Nanoparticles  $\text{TiO}_2$  particles were used as a source materials of Ti where as in electrochemical method Ti path use as a source of Ti. Electrochemical process is room temperature and required low current but in hydrothermal process required high temperature. Here we report electrochemical route for the synthesis of  $\text{BaTiO}_3$ . The electrochemical route is of considerable interest because of a possibly precise particle size control achieved by adjusting current density and applied potential. It has several advantages over both conventional solid-state reaction and liquid phase processes. In this paper, we demonstrate an electrochemical route to synthesis  $\text{BaTiO}_3$  nanowires and its structural, compositional and optical properties were studied.

## 2. EXPERIMENTAL DETAILS

### 2.1. Synthesis of $\text{BaTiO}_3$

The synthesis method has been employed a modified version of the originally used by Chien-Jung Huang et al. [11] for metal particles (in a one-phase electrochemical system). The

schematic diagram of the synthesis system in the present study is shown in figure 1. Two titanium plates were employed as an anode and cathode. The distance between the plates were 2 cm and kept fixed. In the experiment, laboratory grade titanium sheet ( $1\text{ cm} \times 1\text{ cm}$ ) as an anode and cathode were used. A 250 ml Teflon beaker was used as an electrolytic tank. The distilled water for the preparation of the electrolyte was boiled for 1h to remove  $\text{CO}_2$ . The typical electrolytic solution consisted of 60 ml distilled water, 90 ml absolute ethanol, 0.005-mol  $\text{Ba}(\text{OH})_2 \cdot 8\text{H}_2\text{O}$  and 1.0-mol KOH.



**Figure 1.** Synthesis of  $\text{BaTiO}_3$  nanowires by electrochemical route

The electrochemical processes were performed using a DC supply (GPS-30D, 0-30V, 0-5A) with magnetic stirring. Electrolysis was carried out at room temperature, under nitrogen gas for one hour in constant current mode. At this time, the formation of black precipitate was observed. The obtained samples were filtered and washed by 2.0 M  $\text{NH}_4\text{Cl}$  solution to remove the trace unexpected  $\text{BaCO}_3$  and then washed by distilled water and absolute ethanol for several times. Particles were separated by centrifugation and washed at least three times with acetonitrile.

They were dried at room temperature. The nanoparticles samples can be stored at room temperature in the form of a powder and can be re-dispersed in organic solvents like acetonitrile for further characterization.

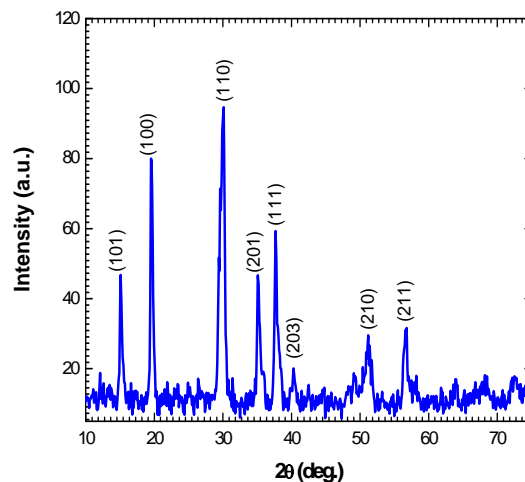
## 2.2. Characterization

The structural properties of the  $\text{BaTiO}_3$  nanoparticles were measured by using a Bruker D8 Advance X-ray diffractometer with  $\text{CuK}\alpha$  radiation of wavelength  $\lambda=1.54056 \text{ \AA}$ . X-ray diffraction (XRD) measurements were carried out in the locked coupled mode in the  $2\theta$  range of  $10\text{-}75^\circ$ . The surface morphology and composition of  $\text{BaTiO}_3$  nanowires were investigated using an 'EVO LS 15' scanning electron microscope (SEM) developed by Carl Zeiss with accelerating voltage of  $\sim 22 \text{ keV}$  and probe current of  $\sim 800 \text{ mA}$ . The FTIR absorption spectra were recorded on a Perkin-Elmer GX FTIR system was used to obtain  $16 \text{ cm}^{-1}$  resolution spectra in the range  $500$  to  $4000 \text{ cm}^{-1}$  region, scanned 30 times (absorbance mode), in order to exploit the instrumental built-up noise reduction algorithm. UV-Vis absorption spectrum was recorded by using a UV-Vis spectrophotometer in the photon wavelength range between  $250$  and  $700 \text{ nm}$ . Room temperature PL measurements were carried out from room temperature by employing a  $488 \text{ nm}$  line of an argon ion laser. The PL spectra were collected on a Jobin Yvon-Horiba Triax 190 spectrometer with a spectral resolution of  $0.3 \text{ nm}$ , coupled with a liquid nitrogen-cooled CCD detector.

## 3. RESULTS AND DISCUSSION

### 3.1. Structural and Compositional Properties

A typical XRD pattern of the electrochemically synthesized  $\text{BaTiO}_3$  nanoparticles is shown in figure 2.



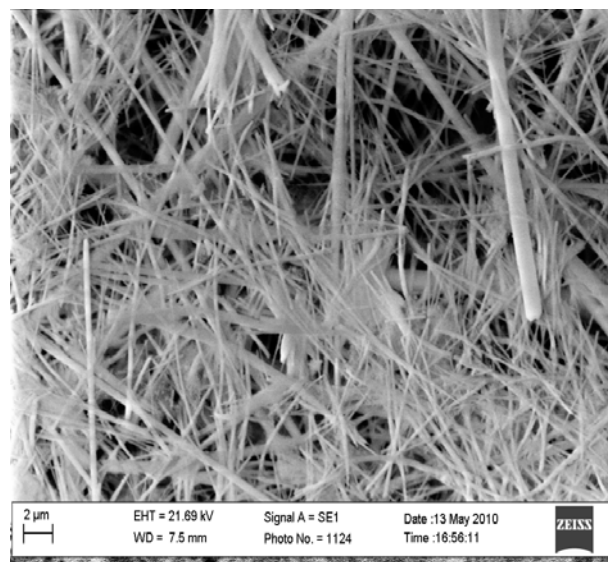
**Figure 2.** XRD pattern of  $\text{BaTiO}_3$  nanoparticles

The diffraction peaks (100), (110), (111), (210), (211) can be indexed to the cubic structured  $\text{BaTiO}_3$  with lattice constant is  $a = 4.031 \text{ \AA}$ , which is in good agreement with the reported data (JCP2.2CAa:31-0174).

Other diffraction peaks (101), (201), (203) are well consistent with hexagonal phase, which had been confirmed by literature (JCP2.2CA:34-0129). It is clear that the (110) peak is sharper and stronger. The width of the nanowires can be determined by using the well-known Scherrer's formula [12] that simplified by Bragg's law:

$$B_{\text{crystallite}} = \frac{0.9\lambda}{B \cos \theta} \quad (1)$$

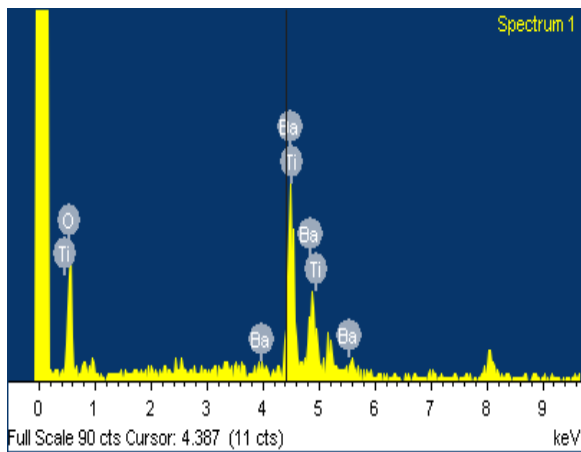
where  $\lambda$  is the wavelength of  $\text{Cu K}\alpha$  radiation, and  $B$  is them full-width at half-maximum (FWHM). The unit of  $B$  should be converted into radian. The width of the  $\text{BaTiO}_3$  nanoparticles was calculated to be  $\sim 15 \text{ nm}$ , which conformed reasonably well to the literature value [10]. This implies that the nanowires are stabilized in the cubic form at room temperature of the  $\text{BaTiO}_3$ . This XRD patterns indicate that  $\text{BaTiO}_3$  was successfully synthesized. Since XRD patterns showed appreciable peak broadening presumable due to the small particle size, which was evidence to the cubic structure.



**Figure 3.** SEM image of BaTiO<sub>3</sub> nanoparticles

Figure 3 shows the SEM image for the surface morphology of electrochemically synthesized BaTiO<sub>3</sub>. The image shows the particles are looks like nanowires in shape, which width is calculated from 11.9 to 62.3 nm. Similar result reported for electrospinning deposited BaTiO<sub>3</sub> nanofiber [13].

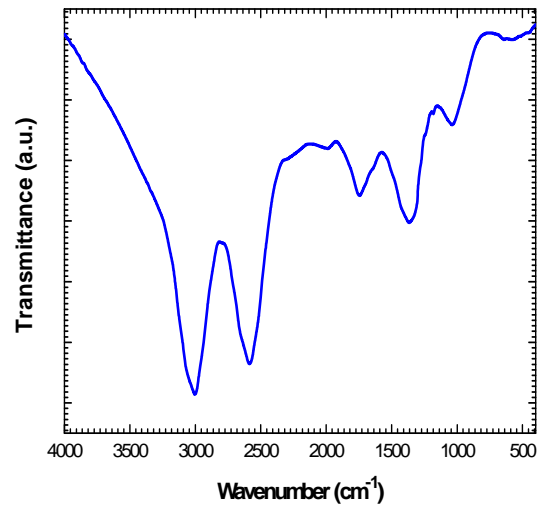
The existence of Ba, Ti, and O elements in synthesized BaTiO<sub>3</sub> nanoparticles were confirmed by the EDX measurement is shown in figure 4. Compositions at three different places on this sample were determined. They show reasonably identical values ( $\pm 0.01$  at. %) that ascertain the compositional uniformity of the sample. The elemental atomic composition shows 16.02%, 5.48% and 78.5% for Ba, Ti and O, respectively.



**Figure 4.** EDX spectrum of BaTiO<sub>3</sub> nanoparticles

### 3.2. Optical properties

Figure 5 shows the FTIR spectrum of the electrochemically synthesized BaTiO<sub>3</sub> nanoparticles.

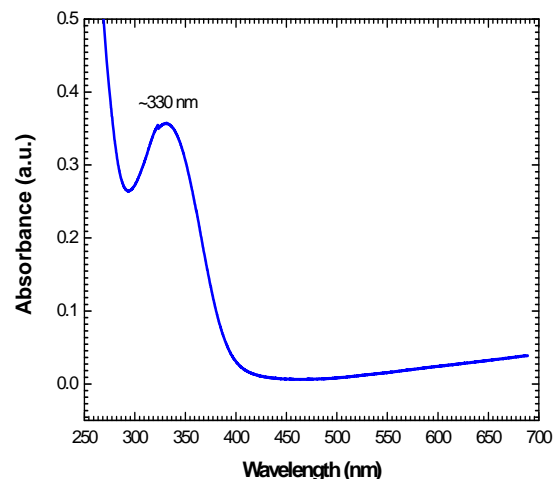


**Figure 5.** FTIR spectrum of BaTiO<sub>3</sub> nanoparticles

FTIR spectrum displays several types of vibrations at 1100, 1350, 1750, 2580 and 3000 cm<sup>-1</sup> and they were assigned to the Ti-O stretching, Ti-OH and Ba-OH, respectively. A broad band centered at 3000 cm<sup>-1</sup> and another strong band at 2580 cm<sup>-1</sup> was assigned to the stretching and in-plane deformation mode of OH, respectively. Several vibrations due to various modes of C-N of the C-NH<sub>2</sub> and N-H of NH<sub>2</sub> were also observed at 1100, 1350 and 1750 cm<sup>-1</sup>, respectively. Therefore, the comparative study of the FTIR spectra further supports the enhancement of redispersibility of the polycrystalline BaTiO<sub>3</sub> nanoparticles. Figure 6 shows the room temperature UV-Vis absorption spectrum of electrochemically synthesized BaTiO<sub>3</sub> in the 200 to 700 nm wavelength range. The nature of the exponential edge and tails may be associated with localized energy states in the band gap. In the high-energy region of the absorption curve, the optical energy band gap is related to the absorbance and to the photon energy by the following equation:

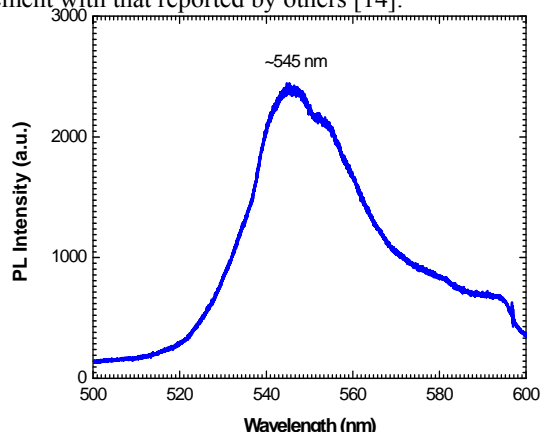
$$h\nu\alpha = (h\nu - E_g^{opt})^2 \quad (2)$$

where  $h$  is the Planck constant,  $\nu$  is the frequency and  $E_g^{opt}$  is the optical band gap. Band gap energy of BaTiO<sub>3</sub> was calculated to be  $\sim 3.1$  eV.



**Figure 6.** UV-Vis absorption spectrum of BaTiO<sub>3</sub> nanoparticles

This result shows that our data is consistent with interpretation that the exponential optical absorption edge and the optical band gap are controlled by structural defects in the BaTiO<sub>3</sub>. This phenomenon showing its good optical quality is in good agreement with that reported by others [14].



**Figure 7.** PL spectrum of BaTiO<sub>3</sub> nanoparticles

Photoluminescence spectrum of BaTiO<sub>3</sub> nanoparticles excited with 488 nm laser source at room temperature presented in figure 7.

PL spectrum of BaTiO<sub>3</sub> nanoparticles have defect related deep level emission in the visible range. The emission in the visible is attributed to the recombination of electrons and holes in the delocalized states. This delocalized state is due to the intrinsic structural defect related to nanocrystal symmetric Ti<sup>3+</sup> in the nanophase barium titanate. The PL is directly related to the exponential optical edge and tail. We believe that the PL observed in this BaTiO<sub>3</sub> nanoparticles arises from a radiative recombination between trapped electrons and trapped holes in tail states. It is obvious that a broad emission band is located at ~545 nm (~2.275 eV) with a shoulder peak at ~557 nm (~2.226 eV), which is a typical oxygen vacancy emission [15]. In this work, the oxygen vacancies in BaTiO<sub>3</sub> nanoparticles act as an emission source under the photo-excitation and result in the PL behavior.

## 4. CONCLUSIONS

BaTiO<sub>3</sub> nanoparticles have been successfully grown by electrochemical route and studied its structural, compositional and optical properties. From these studies it has been assessed that BaTiO<sub>3</sub> was successfully synthesized. The structural and optical measurements reveal that the grown samples are in nanoparticles and its optical properties are well agreements with literature.

## 5. ACKNOWLEDGEMENTS

Authors would like to thank Islamic University, Kushtia, Bangladesh authority for providing funds under the University Research Project to carry out this work.

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